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New Material System for 3rd Generation IR Applications

by Gregory Brill and Yuanping Chen

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New Material System for 3rd Generation IR Applications

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Sensors and Electron Devices Directorate, ARL

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14. ABSTRACT

Much progress has been made in developing high quality mercury cadmium telluride (HgCdTe)/silicon (Si) for large area long wavelength infrared (LWIR) focal plane array (FPA) applications. However, even with all the material advances made to date, there is no guarantee that this technology will be mature enough to meet FPA specifications for a fielded system. With this in mind, the U.S. Army Research Laboratory (ARL) has begun investigating mercury cadmium selenide (HgCdSe) for infrared (IR) applications. Analogous to HgCdTe, HgCdSe is a tunable semiconductor that can detect any wavelength of IR radiation through control of the alloy composition. Additionally, several mature, large area bulk III-V substrates are nearly lattice matched to HgCdSe, giving this system a possible advantage over HgCdTe, for which no scalable, bulk substrate technology exists. We have initiated a study of the growth of HgCdSe using molecular beam epitaxy (MBE). Growth temperature and material flux ratios were varied to ascertain the best growth conditions. Smooth surface morphology has been achieved using a growth temperature much lower than HgCdTe (~80 °C lower). Additionally, no void defects were nucleated at these lower temperatures. Results suggest a linear relationship between the Se/Cd flux ratio and the cut-off wavelength.

15. SUBJECT TERMS

HgCdSe, MBE, GaSb, infrared, II-VI

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1. Objective

The objective of this project was to lay the groundwork for the development of a new tunable II-VI infrared (IR) material system using mature III-V semiconductors as lattice-matched substrates. Specifically, we grew and studied mercury cadmium *selenide* (HgCdSe) as an alternative to mercury cadmium *telluride* (HgCdTe) as an IR-detecting material. The advantage of the HgCdSe system is twofold. First, it is tunable to any IR wavelength of interest by controlling the cadmium (Cd) composition, and second, it is lattice matched to mature III-V semiconductor systems, such as gallium antimonide (GaSb) and indium arsenide (InAs). By using a lattice-matched mature substrate technology, in principle, HgCdSe can be grown with limited generation of dislocations resulting in extremely high quality and uniform IR sensing material, which would greatly improve operability of the focal plane array (FPA). This is completely analogous to HgCdTe grown on lattice-matched bulk cadmium zinc telluride (CdZnTe) substrates, which result in two orders of magnitude reduction in dislocation density over HgCdTe grown on silicon (Si) substrates. The main difference is that CdZnTe substrates are not available in large sizes and are only provided by a sole foreign supplier. In contrast, GaSb substrates are available in 4-in wafer sizes and several U.S. suppliers exist.

2. Approach

2.1   Background

We leveraged our expertise in growing the II-VI IR detecting compound, HgCdTe, by molecular beam epitaxy (MBE) to develop a completely new IR sensing II-VI system, HgCdSe. Currently, HgCdTe is the material used in the majority of fielded Army IR systems and much effort has been expended to push the technology to both large format and low-cost systems while still maintaining superior performance. However, this technology has been sitting at a roadblock for several years that, to date, has not been overcome and no solution appears imminent. Namely, in order to achieve large format sizes as required for 3rd generation, a new Si-based composite substrate technology needed to be developed since current lattice-matched substrate technology (bulk grown CdZnTe) is severely limited in size and scalability (a maximum of 6 x 6 cm^2 is commercially available). However, due to the huge lattice mismatch between Si and HgCdTe (19%) and associated strain energy, misfit dislocations need to be generated somewhere in the thin film stack to alleviate this energy which ultimately propagates into the IR-absorbing layer. Generally, two orders of magnitude higher dislocation density is present in scalable HgCdTe/Si material with respect to non-scalable HgCdTe/CdZnTe. It has been demonstrated that this higher dislocation level results in lower device performance (*1*). There is ongoing effort within the community to either reduce dislocation density in HgCdTe/Si or render the dislocations

electrically inactive, but, to date, no single approach appears to offer a clear and immediately successful option. We propose that HgCdSe will act very similarly to HgCdTe in terms of its IR material properties, but will have the great advantage of having commercially available large area (and scalable) substrates readily available. Additionally, our previous work on MBE growth of CdSeTe indicated that the Cd-selenide (Se) bond strength is stronger than the Cd-telluride (Te) bond strength (*2*). We expect this to be another advantage of HgCdSe over HgCdTe in that the material itself may be more tolerant of dislocations due to this fact.

Figure 1 shows the lattice constant and bandgap of various semiconductor systems. As shown in figure 1, mercury selenide (HgSe) is a semimetal and cadmium selenide (CdSe) is a wide bandgap semiconductor, both with nearly identical lattice constants (HgSe = 6.08 A, CdSe = 6.05 A). By forming an alloy of $Hg_{1-x}Cd_xSe$, the bandgap of the material can be tuned to absorb any wavelength of IR light resulting in an ideal material to cover the entire IR spectral range. In addition, two III-V binary semiconductors are available to use as substrates that are nearly lattice matched to HgCdSe, specifically InAs and GaSb. These substrates are developed and readily available from commercial suppliers within the U.S. By using III-V bulk substrates, a starting template for HgCdSe growth with a very low dislocation density will be available. Presently, GaSb is quoted as having a dislocation density of less than 10^4 cm^{-2} (based on data from the MTI Corporation and Wafer Technology). Even with the small lattice mismatch present between GaSb and HgCdSe (f ~0.70% depending on the exact HgCdSe composition), the final HgCdSe dislocation density is expected to measure in the 10^4 cm^{-2} to low 10^5 cm^{-2} range. This type of dislocation density is exactly what is achieved for HgCdTe material grown on bulk CdZnTe substrates and which produces state-of-the-art infrared focal plane arrays (IRFPAs) for numerous Army applications (*3*). There is no reason to assume that achieving HgCdSe material with the same dislocation density value will not yield similar IRFPA performance.
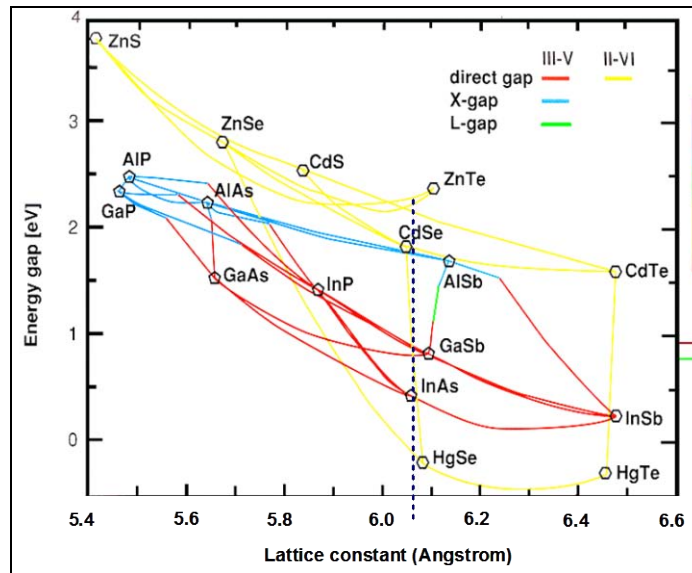

Figure 1. Energy gap versus lattice parameter for several semiconductor material systems.

The IR capability of HgCdSe has been known for some time (*4*). However, due to the major effort spent on developing HgCdTe as the primary material for high resolution IR applications, very little work has been expended on studying the $Hg_{1-x}Cd_xSe$ material system. In fact, only one group has ever grown HgCdSe by MBE and that work was done in 1993 (*5*). Their work used zinc telluride (ZnTe) and CdZnTe as substrates and resulted in only moderate results. However, we feel part of their issue was related to improper substrate cleaning, as much was not known about the proper II-VI substrate surface preparation process for MBE growth 15 years ago. Additionally, this work did not address the use of III-V bulk substrates for HgCdSe growth. Another group has studied the MBE growth of HgSe on GaSb(001) substrates (*6*). This is more relevant to our proposed effort, but still does not address the specific issues associated with the alloy HgCdSe. Additionally, for reasons we describe in section 2.2, the (001) orientation most likely will not be suitable for our efforts.

2.2   Substrate Choice

As alluded to in section 2.1, the main advantage of HgCdSe is that it is nearly lattice matched to a mature III-V bulk substrate technology, specifically GaSb. Clearly, the use of GaSb substrates is a key component of this program. However, in addition to growth on GaSb substrates, we chose to use ZnTe/Si substrates as well. ZnTe is also nearly lattice matched to HgCdSe with a lattice parameter of 6.10 A, making it an excellent candidate for high quality HgCdSe growth. Additionally, by using ZnTe/Si for the growth of HgCdSe, we have a direct comparison to HgCdTe growth, which uses cadmium telluride (CdTe)/Si substrates. This allowed us to jumpstart our effort on HgCdSe by leveraging our expertise in growing HgCdTe material. Both substrates are II-VI compounds containing Te, so the substrate cleaning process (both chemically and thermally) as well as the MBE nucleation process are identical. ZnTe/Si also has the advantage that we can grow these substrates in-house using our II-VI MBE system, meaning we have a quality controlled and unlimited supply of substrates to conduct numerous HgCdSe growth experiments.

Regardless of the substrate choice, initial work used the (211) orientation as this is the preferred orientation for HgCdTe. Again, this was an effort to leverage our expertise with HgCdTe, in which the (211) orientation has been found to minimize twin defects and lead to easier incorporation of mercury (Hg) into the matrix with respect to (001). For these reason, we chose the (211) orientation for our HgCdSe study as well.

Finally, although we are capable of growing on 3-in wafers, we used smaller substrates initially. This was done to minimize costs associated with substrate consumption as we fully expected to conduct numerous growth experiments.

2.3   Experimental Plan

Within the semiconductor community, there exists very limited work on the growth and characterization of HgCdSe material in general and even less using MBE. It was the goal of this

study to develop the basic understanding of the proper conditions to achieve high quality HgCdSe growth by MBE on both GaSb(211) substrates as well as on ZnTe/Si(211) substrates. The main parameter studied was the growth temperature, with HgCdSe material quality assessed by x-ray diffraction and surface morphology characterization (defect formation and overall smoothness). In addition, the constituent material fluxes were varied to study the relationship between cut-off wavelength, which is a function of alloy composition, and the fluxes used during growth.

3. Results

3.1 Substrate Preparation

Since both GaSb(211) and ZnTe/Si(211) substrates were used for HgCdSe growth, two different substrate preparation processes had to be developed to ensure a proper surface for growth nucleation. For ZnTe/Si substrates, we employed our standard cleaning process used for CdTe/Si substrates. This includes a dilute Bromine-methanol etch to remove approximately 0.3 μm of the topmost surface, followed by several methanol rinses, a dilute hydrochloric acid (HCL):de-ionized (DI) water dip to remove any residual oxides and metallic elements, followed finally by a 2–4 min DI rinse. This process removes any contaminants from the surface of the substrate along with the native oxide and leaves a Te-rich surface stoichiometry. Once loaded into the MBE growth chamber, the substrate is heated to remove the excess Te (Te desorbs at ~220 °C). Further heating is conducted to ensure any remaining Te-oxide is desorbed. This final heating process is done under a Te overpressure to ensure no roughening of the surface occurs. This process is monitored in-situ by reflection high energy electron diffraction (RHEED). Once a clean surface is obtained, the substrate is cooled to the desired growth temperature. This substrate cleaning and surface preparation is completely analogous to the cleaning and wafer preparation of CdTe/Si composite substrates and as expected, yielded good surfaces for HgCdSe nucleation, as observed by RHEED.

In contrast, wafer preparation of GaSb substrates was a non-standard process considering our MBE configuration. At present, our II-VI MBE cluster system does not have the appropriate III-V source materials to clean and prepare a GaSb substrate within the same ultra high vacuum (UHV) system that houses our Hg source. Instead, we loaded the "epi-ready" GaSb substrate into a separate III-V MBE system where the native oxide was thermally removed under an antimony (Sb) overpressure at 590 °C. This process was easily observed by RHEED. After which, an approximate 0.2 μm GaSb epitaxial layer was re-grown on the substrate at 500 °C. This is done to ensure a clean and smooth surface is available for HgCdSe nucleation. This is a standard process for GaSb substrates used within the III-V semiconductor community. Normally, after these steps are completed, the substrate would be set to the desired growth temperature and growth would be initiated. However, in our situation, we had to remove the

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substrate from the III-V chamber and load it into the II-VI system. This exposes the surface to atmosphere, which obviates the entire cleaning process as now an oxide sits on the otherwise pristine GaSb surface. This oxide (either gallium [Ga]-oxide or Sb-oxide) is not able to be removed without damaging the substrate surface in the II-VI system. To circumvent this issue, after completion of the GaSb homoepitaxial growth, the substrate was cooled to room temperature where an amorphous arsenic (As) cap layer was deposited. This capping layer was used to protect the GaSb surface during the time the wafer was exposed to atmosphere. This As cap, and the associated oxide (As-oxide), was then thermally removed in the Hg MBE growth chamber. This process was monitored by RHEED as well as x-ray photoelectron spectroscopy (XPS) to determine the chemical species remaining on the substrate surface after completion of the thermal preparation process. Figure 2 shows XPS spectral from three separate GaSb wafers. Figure 2a shows the spectra due to Ga atoms on the surface, while figure 2b shows spectra due to Sb and oxygen (O) atoms on the surface. The topmost (black) spectra in either figure shows the As-capped GaSb surface after loading from the III-V system into the II-VI system. Neither Ga or Sb is observed in either spectra, which is expected as the As cap has successfully buried and protected the GaSb surface. The peak at 531 eV in figure 2b is due to the oxygen present on the surface which accumulates during the substrate's exposure to air. The middle spectra (blue) is taken after thermal evaporation of the As cap in the II-VI MBE system.
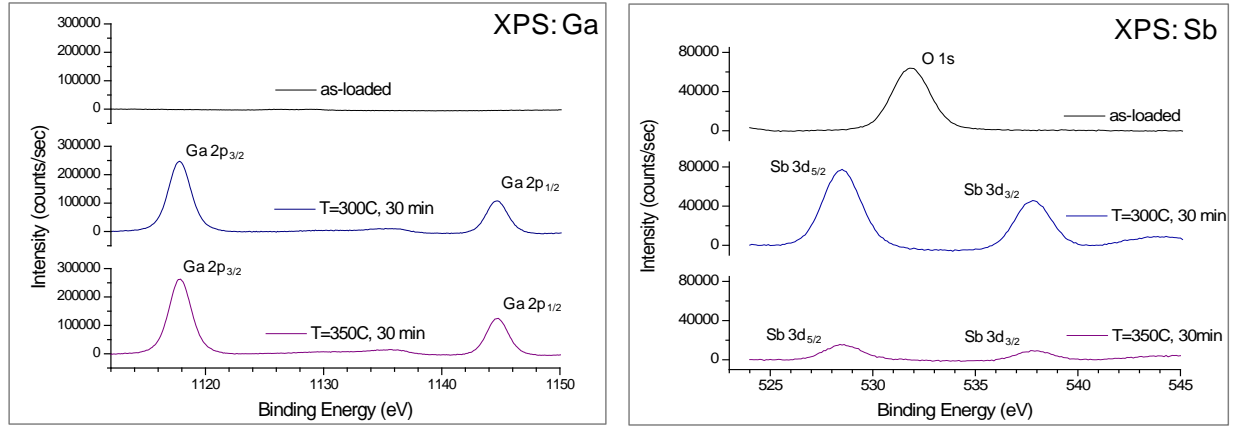

Figure 2. XPS spectra looking at the signal from (a) Ga atoms and (b) Sb and O atoms.

The substrate in this experiment was held at 300 °C for 30 min. This is the temperature at which the RHEED pattern first re-emerged indicating the amorphous As cap was being removed. Note now that the Ga and Sb doublet peaks are clearly observed. The bottom spectra (magenta) is from an As-capped GaSb substrate that was heated to 350 °C for 30 min. The Ga peak intensity is the same as the 300 °C sample, but now the Sb peak intensity is substantially decreased. This indicates that Sb has started to evaporate from the substrate surface leaving a Ga-rich nucleation layer. This is not an ideal nucleation layer for HgCdSe as Ga-Se intermixing at the interface is a concern during the nucleation process. Clearly heating at 350 °C is not ideal. However, even though the spectra from the 300 °C desorption process indicated a stoichiometric GaSb surface,

further analysis using XPS of the As and secondary O peaks indicated that 100% removal of the As and O was not a repeatable process. In some experiments at 300 °C, a sub-monolayer presence of As and O would remain on the surface. The non-repeatability of obtaining a pristine, stoichiometric GaSb surface is most likely due to the variation in the amount of time the substrate was exposed to air as well as the variation in the As cap layer thickness from substrate to substrate.

In summary, the substrate preparation of GaSb substrates is not optimized given our current MBE configuration. The surface can be cleaned in such a manner that HgCdSe nucleation and growth can occur, but a more pristine GaSb surface needs to be obtained to take full advantage of the benefits related to a bulk grown, high quality substrate. The fact that we cannot go from a III-V system, where a GaSb homoepitaxial layer can be grown, directly into the II-VI Hg system without exposing the sample to air is a limitation. In contrast, the ZnTe/Si substrate preparation process is optimized as it mimics our work on developing and working with CdTe/Si composite substrates. However, in either case, HgCdSe growth experiments were conducted and material characterized.

3.2 HgCdSe Material Properties

HgCdSe growth was conducted using elemental Hg, Cd, and Se sources. The Se and Hg sources are valved sources while the Cd source is a standard Knudsen effusion cell. Nominal flux values were Hg $\sim 1 \times 10^{-3}$, Cd $\sim 4 \times 10^{-7}$, and Se $\sim 4 \times 10^{-6}$ Torr as measured by a nude ion gauge located at the substrate growth position. These are similar fluxes used for the growth of long wavelength infrared (LWIR) HgCdTe, replacing Se for Te. These flux levels would result in a HgCdTe growth rate of ~ 2 µm/hr. For this study, the growth fluxes were kept constant while the growth temperature was varied. The goal was to identify the appropriate growth temperature for HgCdSe and study the surface morphology, crystallinity, and defect structure of the films.

Since epitaxial growth of Hg-based compound is very sensitive to the growth temperature due to the extremely low Hg sticking coefficient, one way to examine the effect of growth temperature is by looking at the growth rate under fixed material fluxes. Figure 3 shows the HgCdSe growth rate as a function of growth temperature. The growth rate is constant, averaging around 1.6–1.7 µm/hr, for growth conducted at 130 °C or below. Once the growth temperature was raised above 130 °C, the growth rate dropped off dramatically. This indicates that the sticking coefficients of either Hg and/or Se drop significantly at these higher growth temperatures and places an upper limit for temperature based on the fluxes used in these experiements. Also, note that the optimal HgCdTe growth temperature is 185 °C, using analogous fluxes, implying that replacing Te for Se induces a change in the growth kinematics between HgCdTe and HgCdSe.
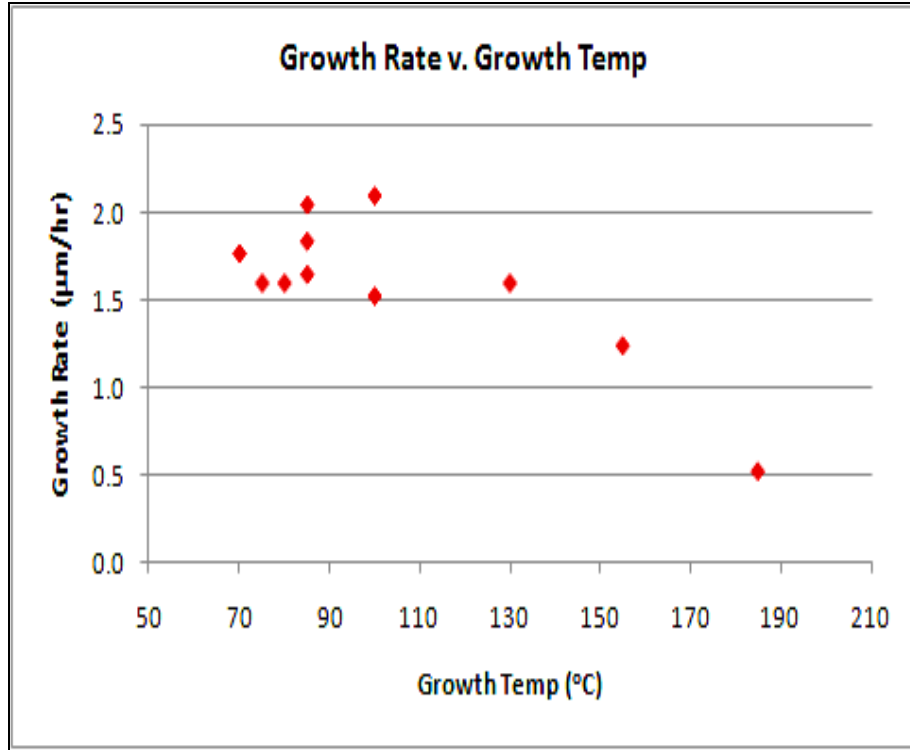
Figure 3. HgCdSe growth rate as a function of growth temperature.

Next we examined the surface morphology of the HgCdSe epilayers, which is one of the key metrics for the crystalline quality of any heteroepitaxial layer and suitability for device fabrication. The results are illustrated in figure 4. For HgCdSe grown at 75 °C, the surface morphology is extremely rough. Furthermore, RHEED monitoring during growth at this temperature indicated that growth initiated in a crystalline manner, but eventually evolved into an amorphous film growth. This was evidenced through the RHEED evolving from a streaky RHEED pattern (crystalline) to a spotty RHEED pattern (three-dimensional surface features) to finally a very dim ring pattern that was independent of azimuth (amorphous). Furthermore, no x-ray diffraction could be obtained for the [422] peak, another indication of amorphous growth. This data sets a lower bound for HgCdSe growth.
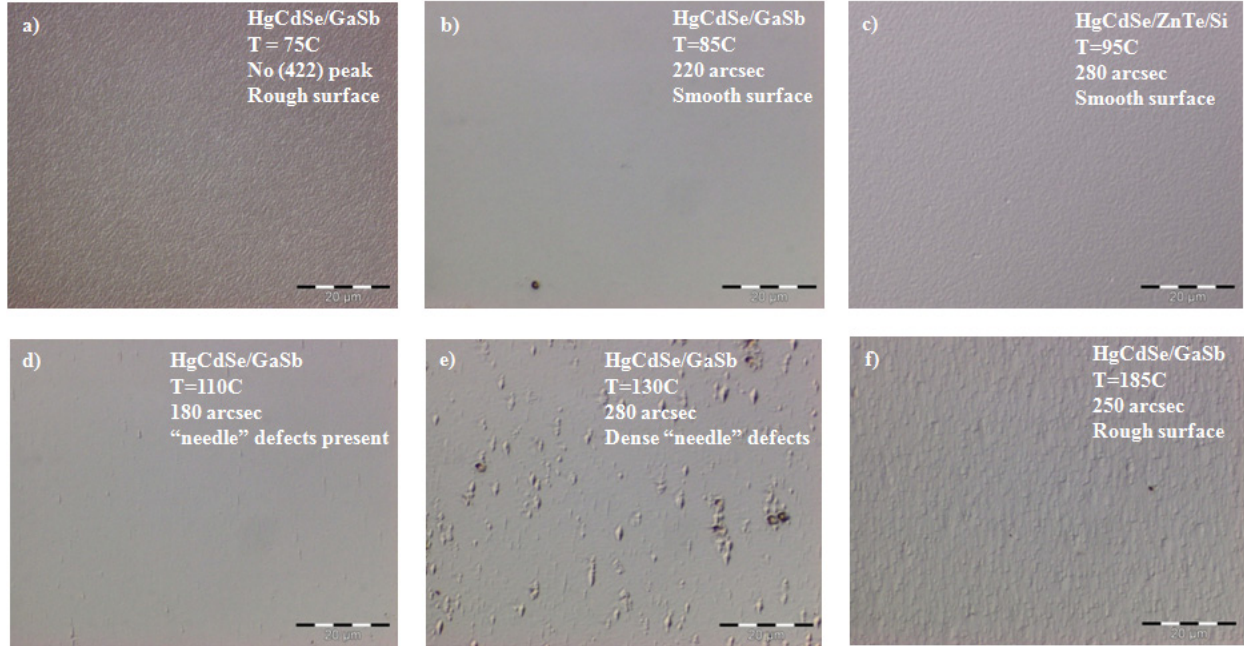
Figure 4. Surface images taken with an optical microscope of HgCdSe layers grown at different temperatures. All layers were nucleated on GaSb substrates, except the image shown in figure 4c, which was nucleated on a ZnTe/Si substrate. The "orange peel" surface morphology in this image is in artifact of the substrate surface morphology.

At temperature between 80–100 °C, HgCdSe growth appeared optimal. The RHEED pattern remained streaky throughout growth and the surface morphology mimicked that of the underlying substrate. For nucleation on GaSb, this resulted in extremely smooth surfaces. For nucleation on ZnTe/Si substrates, this resulted in an "orange peel" type surface as the ZnTe/Si surface itself was not entirely smooth. Note, that optimizing ZnTe/Si material growth was not an emphasis of this initial study. However, it will be a point of emphasis as this program progresses. Additionally, very little defects were observed on these surfaces, regardless of substrate choice. X-ray diffraction measurement resulted in HgCdSe[422] peaks with full width at half maximum (FWHM) values of 220 arcsec for growth on GaSb and 280 arcsec for growth on ZnTe/Si. In general, the smaller the peak width, as measured by x-ray FWHM, the more perfect the crystalline material. It should be noted that all layers grown in this study measured ~4 μm in thickness unless otherwise noted.

As the growth temperature was increased, the x-ray FWHM was observed to decrease. Growth on GaSb and ZnTe/Si resulted in layers with x-ray FWHM of 180 and 250 arcsec, respectively. However, "needle" defects began to appear on the surface as observed in figure 4d and 4e. The defect size and density increased as the growth temperature was increased. Additionally, the appearance of these defect structures is independent on the choice of substrate. Nucleation on both GaSb and ZnTe/Si resulted in similar surface morphologies indicating that (1) these defects are due to HgCdSe growth conditions and (2) that although not optimized, the GaSb substrate preparation process is adequate for good growth. It is interesting to note that even though the

overall growth rate remains constant, a detrimental surface morphology is obtained at temperatures above 100 °C.

As the temperature is increased further above 130 °C, the surface appears extremely rough and the x-ray FWHM broadens. In essence, the entire surface is nucleated with such a high density of needle defects that these defects are overlapping, leaving a very rough, very facetted surface. Clearly, nucleation is occurring far away from the optimal conditions at this temperature given the fluxes used.

Finally, one surprising finding is that the HgCdSe grown on either GaSb or ZnTe/Si substrate does not exhibit classic void defects. These defects, which are quite common on HgCdTe layers, are large crater-like defects measuring 5–6 µm in diameter. Since these defect sizes are on the same order of magnitude as the pixel dimensions (nominally 20 µm) of an IRFPA, they act as killer defects and severely affect IRFPA operability. A comparison of surface morphologies between HgCdTe and HgCdSe is shown in figure 5. One possible explanation for the lack of void formation is that Se tends to have much higher vapor pressure than Te, and therefore a higher surface mobility even at such low growth temperatures, which prevent the formation of Se clusters. It is speculated that this absence of Se clusters removes the seed site for a void defect, as opposed to HgCdTe nucleation in which Te clusters can easily form and have been linked to void defect formation (7).
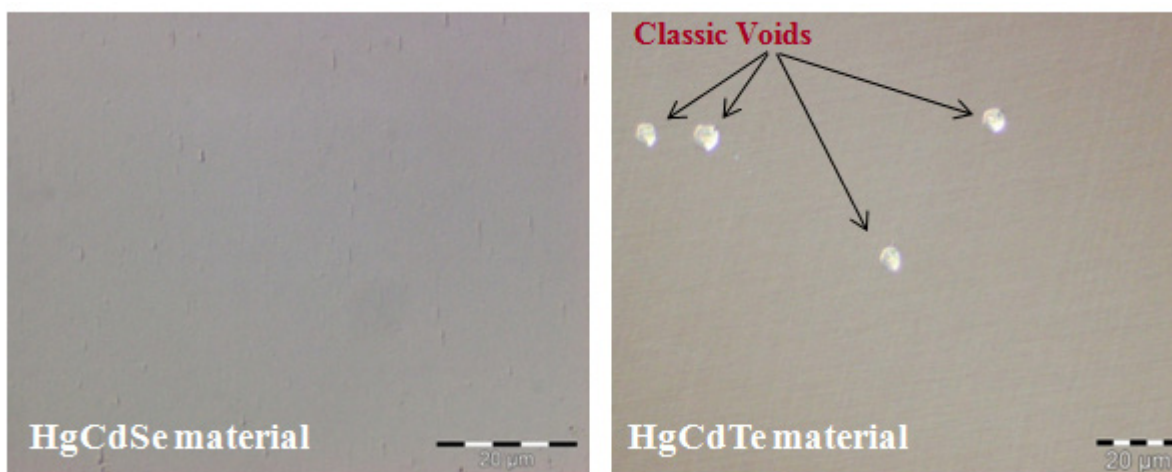

Figure 5. Surface morphology of HgCdSe and HgCdTe showing typical defects in either material. No void defects were observed for HgCdSe material for growth at any temperature.

Finally, to ensure HgCdSe material is indeed tunable, we varied the material fluxes and measured the cut-off wavelength of the layer. Figure 6 shows several films characterized by Fourier Transform Infrared Spectroscopy (FTIR) that clearly show that the material composition, and hence its IR absorbing properties, is tunable. Figure 7 shows the relationship between the flux properties used during growth and the material composition. A linear relationship exists

between Se/Cd flux ratio and the layer composition. This fact makes it quite easy to tailor HgCdSe material to the appropriate IR wavelength of interest.
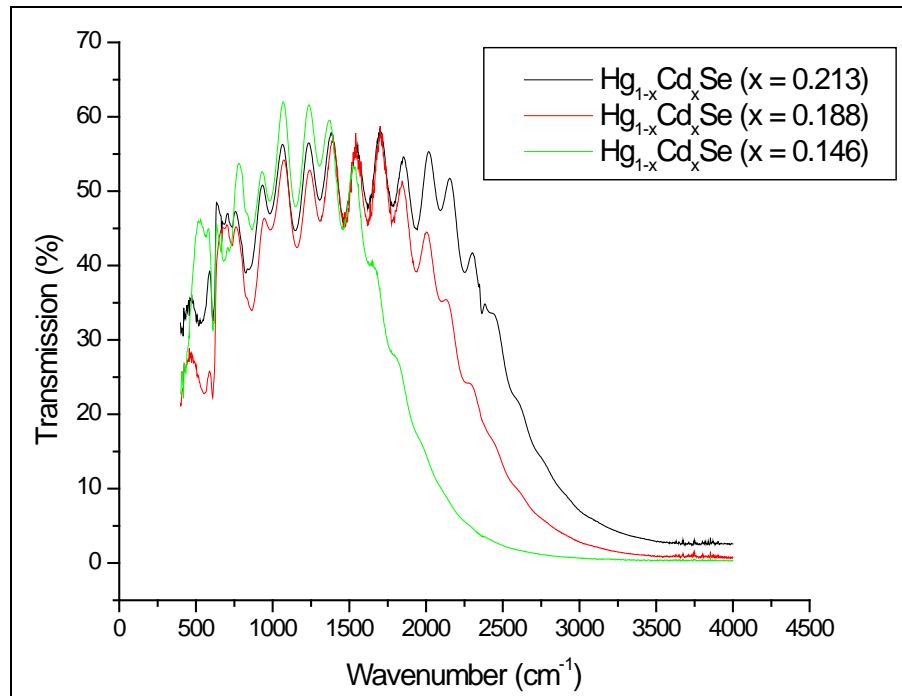

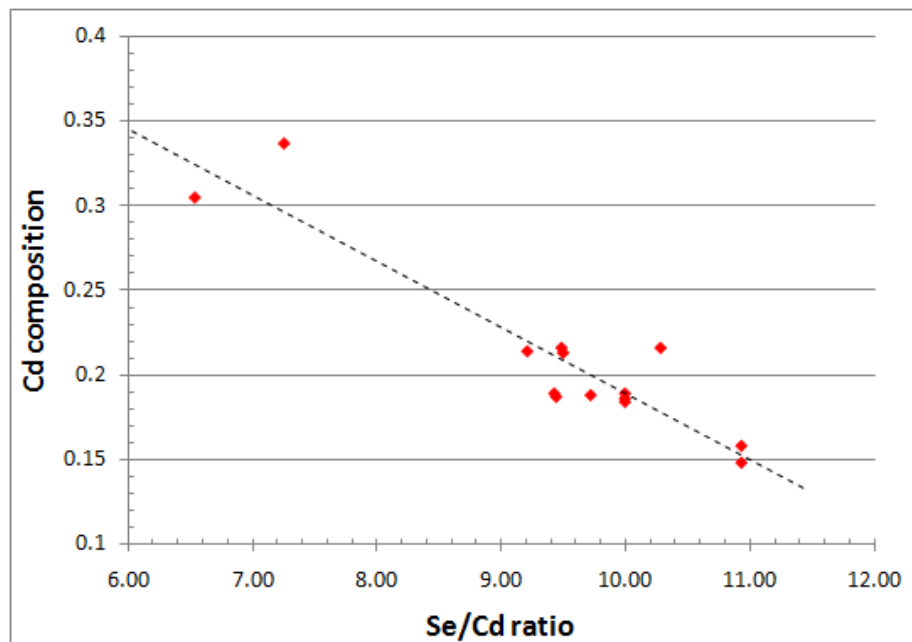Figure 6. FTIR spectra from HgCdSe layers with different Cd compositions.

Figure 7. Relationship between Se/Cd flux ratio used during MBE growth and the
resulting HgCdSe composition.

4. Conclusions and Future Work

HgCdSe is a promising IR material that can be grown on scalable substrates. To date, very little information exists on the growth and material properties of HgCdSe grown by MBE. In this study, we have grown HgCdSe on both GaSb(211) and ZnTe/Si(211) substrates. This is the first demonstration of HgCdSe material grown on GaSb substrates. Although the surface preparation process for each substrate differs substantially, the HgCdSe growth is quite similar, only dependent on the growth temperature and material fluxes. We have determined the growth temperature window for HgCdSe material growth given a set of material fluxes and demonstrated HgCdSe material with smooth surface morphology and good crystallinity as determined by x-ray diffraction. Additionally, we have demonstrated that the material is tunable within the IR spectral range by controlling the Se/Cd flux ratio during growth.

Many more experiments need to be conducted to fully explore the growth dynamics of HgCdSe material by MBE. Specifically, the process of needle defect formation needs to be understood and a more detailed analysis of the lack of void defect formation needs to be obtained to fully exploit this attribute. Additionally, further research needs to be undertaken to better prepare the GaSb(211) surfaces for HgCdSe growth. This work will be carried out in conjunction with our collaborators at Texas State University, a subcontractor on the FY11 DRI effort.

In addition to the research planned to study MBE growth parameters, annealing studies and electrical studies will be carried out as an understanding and control of the electrical properties of HgCdSe are paramount for device performance.

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6. Transitions

Through the research conducted under this Director Research Initiative (DRI) program, we have gained outside funding for continued work in developing HgCdSe material for IR applications. The Air Force Office of Scientific Research (AFOSR) is funding a five-year collaboration between the U.S. Army Research Laboratory (ARL) and Arizona State University titled "Optically biased monolithically integrated multicolor photodetectors." This program is designed to develop materials, including HgCdSe, for multiband (ultraviolet [UV] to IR) sensor capabilities within a single pixel. This program started in July 2010 and we presented our HgCdSe research at the official kick-off meeting in November 2010. ARL is receiving $75K/year.

The U.S. Army Research Office (ARO) has recently funded Texas State University to study HgCdSe material for IR applications. Texas State University is receiving $50K/year in funding and a graduate student will be dedicated to this research. This program began in July 2010 and is being monitored closely by the principal investigator (PI) of this DRI program to ensure the best use of resources and a highly coordinated effort is undertaken between ARL and Texas State University. In addition, Texas State University has a congressional program for the Center for Heterofunctional Materials (CHM), which is also managed by ARL. The CHM is comprised of four tasks, including the development and study of HgCdSe material.

We have presented a paper at the 2010 U.S. Workshop on the Physics and Chemistry of II-VI Materials and submitted a corresponding manuscript for publication. This is the first work in the community regarding HgCdSe growth on scalable GaSb substrates.

Finally, this work is continuing under the DRI program as the second year of this project was selected for funding in FY11.

List of Symbols, Abbreviations, and Acronyms

AFOSR	Air Force Office of Scientific Research
arcsec	arcseconds
ARL	U.S. Army Research Laboratory
ARO	U.S. Army Research Office
As	arsenic
Cd	cadmium
CdSe	cadmium selenide
CdTe	cadmium telluride
CdZnTe	cadmium zinc telluride
CHM	Center for Heterofunctional Materials
DI	de-ionized
DRI	Director's Research Initiative
FPA	focal plane array
FTIR	Fourier Transform Infrared Spectroscopy
FWHM	full width at half maximum
Ga	gallium
GaSb	gallium antimonide
HCL	hydrochloric acid
Hg	mercury
HgCdSe	mercury cadmium selenide
HgCdTe	mercury cadmium telluride
HgSe	mercury selenide
InAs	indium arsenide
IR	infrared

IRFPA	infrared focal plane array
LWIR	long wavelength IR
MBE	molecular beam epitaxy
O	oxygen
PI	principal investigator
RHEED	reflection high energy electron diffraction
Sb	antimony
Se	selenium
Si	silicon
Te	tellurium
UHV	ultra high vacuum
UV	ultraviolet
XPS	x-ray photoelectron spectroscopy
ZnTe	zinc telluride